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ISO 10304-4

Second edition
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Water quality — Determination of dissolved anions by liquid chromatography of ions —

Part 4: Determination of chlorate, chloride and chlorite in water with low contamination

Qualité de l'eau — Dosage des anions dissous par chromatographie des ions en phase liquide —

Partie 4: Dosage des ions chlorate, chlorure et chlorite dans des eaux faiblement contaminées

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 230, *Water analysis,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 10304-4:1997), which has been technically revised. The main changes compared to the previous edition are as follows:

— in the introduction, all requirements concerning the application of the method have been deleted and moved to other clauses;

— in Clause 2, all the references made but withdrawn since the publication of the 1997 edition (e.g. ISO 10304-2) have been deleted and the references ISO 5667-1 and ISO 5667-3 have been moved to the Bibliography;

— in 6.8, various eluent formulations have been reduced to one example;

— in Clause 8, the calculation procedure for the peak resolution according to the USP definition [Formula (1)] has been completed with the EP definition [Formula (2)] (both calculations are equivalent);

— in 9.1, information that drinking water disinfection treatment using chlorine dioxide can cause the formation of chlorite and chlorate (paragraph 2) and helpful precautions to minimize/eliminate such formation (paragraph 3) have been added;

— in Clause 11, the option to report result concentrations in microgram per litre has been added.

A list of all parts in the ISO 10304 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Water quality — Determination of dissolved anions by liquid chromatography of ions —

Part 4: Determination of chlorate, chloride and chlorite in water with low contamination

WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices.

IMPORTANT — It is absolutely essential that tests conducted in accordance with this document be carried out by suitably qualified staff.

1 Scope

This document specifies a method for the determination of the dissolved anions chlorate, chloride and chlorite in water with low contamination (e.g. drinking water, raw water or swimming pool water).

The diversity of the appropriate and suitable assemblies and the procedural steps depending on them permit a general description only.

For further information on the analytical technique, see Bibliography.

An appropriate pre-treatment of the sample (e.g. dilution) and the use of a conductivity detector (CD), UV detector (UV) or amperometric detector (AD) make the working ranges given in Table 1 feasible.

Table 1 — Working ranges of the analytical method

Anion	Working range mg/l[a]	Detection
Chlorate	0,03 to 10	CD
Chloride	0,1 to 50	CD
Chlorite[b]	0,05 to 1	CD
	0,1 to 1	UV; λ = 207 nm to 220 nm
	0,01 to 1	AD; 0,4 V to 1,0 V

[a] The working range is restricted by the ion-exchange capacity of the columns. If necessary, samples can be adjusted to this range by dilution.

[b] The minimum working range for chlorite of 0,05 mg/l was obtained using calibration checks, but the interlaboratory trials (see Table A.4) showed that it is difficult to obtain this with sufficient accuracy, and only if taking great care.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 8466-1, *Water quality — Calibration and evaluation of analytical methods — Part 1: Linear calibration function*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Interferences

Organic acids such as mono- and dicarboxylic acids or disinfection by-products (e.g. chloroacetic acid) can interfere.

Dissolved organics can react with the working electrode of the amperometric detector, causing a decrease in sensitivity.

The presence of fluoride, carbonate, nitrite and nitrate can cause interference with the determination of chlorate, chloride and chlorite. The respective concentrations given in Table 2 are typical for conductivity, UV and amperometric detectors.

Elevated loads of chloride and bromide can cause interference with the determination of chlorite and chlorate. Remove chloride and bromide with the aid of special exchangers (9.2).

Solid particles and organic compounds (such as mineral oils, detergents and humic acids) shorten the lifetime of the separator column. They are therefore eliminated from the sample prior to analysis (Clause 9).

Table 2 — Typical cross-sensitivity of anions

Relation of the mass concentration[a] of		Detection method
measured ion	interfering ion	
1 part chlorate	50 parts bromide	CD
1 part chlorate	500 parts nitrate	CD
1 part chloride	500 parts fluoride	CD
1 part chloride	1 000 parts chlorite	CD
1 part chloride	50 parts nitrite	CD
1 part chlorite	100 parts fluoride	CD
1 part chlorite	10 parts fluoride	UV
1 part chlorite	1 000 parts carbonate	CD
1 part chlorite	1 000 parts chloride	CD / UV / AD
1 part chlorite	100 parts nitrite	AD

[a] In case the quality requirements in Clause 8 (e.g. see Figures 2 and 3) are not achieved, the sample shall be diluted.

5 Principle

Liquid chromatographic separation of chlorate, chloride and chlorite is carried out by means of a separator column. A low-capacity anion exchanger is used as the stationary phase and usually aqueous solutions of salts of weak mono- and dibasic acids as mobile phases (eluent, 6.8).

Detection is by CD with or without suppressor device, UV or AD.

When using conductivity detectors, it is essential that the eluents have a sufficiently low conductivity. For this reason, conductivity detectors are often combined with a suppressor device (cation exchangers)

which reduces the conductivity of the eluent and transforms the sample species into their respective acids.

UV detection measures the absorption directly or indirectly.

Amperometric detection of chlorite is carried out via measurement of the current generated by the oxidation of chlorite. The oxidation voltage for chlorite depends on the pH of the eluent. The use of carbon electrodes has proved successful.

The concentration of the respective anions is determined by a calibration of the overall procedure. Particular cases may require calibration by means of standard addition (spiking). Control experiments are necessary to check the validity of the calibration function. Replicate determinations can be necessary.

6 Reagents

Use only reagents of recognized analytical grade. Carry out weighing with an accuracy of 1 % of the nominal mass. An increase in electrical conductivity due to an uptake of carbon dioxide does not interfere with the determination. Use and prepare alternative concentrations or volumes of solutions as described below, if necessary. Alternatively, use commercially available solutions of the required specification.

6.1 Water.

The water used shall have a resistivity ≥18 MΩ·cm (25 °C) and shall not contain particulate matter of a particle size >0,45 µm.

6.2 **Sodium hydrogencarbonate**, $NaHCO_3$.

6.3 **Sodium carbonate**, Na_2CO_3.

6.4 **Sodium hydroxide solution**, *c*(NaOH) = 0,1 mol/l.

6.5 **Sodium chlorite**, $NaClO_2$ (80 %).

6.6 **Sodium chloride**, NaCl.

6.7 **Sodium chlorate**, $NaClO_3$.

6.8 Eluents.

Degas all eluents used. Take steps to avoid any renewed air pick up during operation (e.g. by helium sparging, inline degasser).

The choice of eluent (e.g. based on sodium carbonate or sodium hydroxide solutions, potassium hydroxide, mixed with organic modifiers, if needed) depends on the choice of column and detector; seek advice from the column supplier. Apply eluents that were prepared: manually, by inline dilution or electrochemically *in situ*. The chosen combination of separator column and eluent shall conform to the resolution requirements stated in Clause 7. Use eluents as long as the requirement in Clause 8 is met.

An example for an appropriate eluent manually prepared is given in 6.8.2.

6.8.1 Sodium carbonate/sodium hydrogencarbonate concentrate.

For the eluent concentrate preparation:

— Place 19,1 g of sodium carbonate (6.3) and 14,3 g of sodium hydrogencarbonate (6.2) into a volumetric flask of nominal capacity 1 000 ml, dissolve in water (6.1) and dilute to volume with water (6.1).

— The solution contains 0,18 mol/l of sodium carbonate and 0,17 mol/l of sodium hydrogencarbonate. This solution is stable for several months if stored at 2 °C to 6 °C.

6.8.2 Sodium carbonate/sodium hydrogencarbonate eluent.

The following eluent is applicable for the determination of chlorate, chloride and chlorite:

— Pipette 50 ml of the sodium carbonate/sodium hydrogencarbonate concentrate (6.8.1) into a volumetric flask of nominal capacity 5 000 ml and dilute to volume with water (6.1).

— The solution contains 0,001 8 mol/l of sodium carbonate and 0,001 7 mol/l of sodium hydrogencarbonate. Store the solution in amber-coloured glass and renew it every 3 d.

6.9 Stock solutions.

Prepare stock solutions of concentration ρ = 1 000 mg/l for each of the anions chlorate, chloride and chlorite.

Dissolve the appropriate mass of each of the substances (6.5, 6.6 and 6.7), prepared as stated in Table 3, in approximately 800 ml of water (6.1, degassed with nitrogen or helium), in volumetric flasks of nominal capacity 1 000 ml, add 1 ml of sodium hydroxide solution (6.4). Dilute the volume with water (6.1). The solutions are stable as indicated in Table 3.

Alternatively, use commercially available stock solutions of the required concentration.

Table 3 — Mass of portion, pre-treatment and storage suggestions for stock solutions

Anion	Compound	Concentration derived from substance-portion g/l	Pre-treatment	Storage
Chlorate	$NaClO_3$	1,275 3 ± 0,013	Dry in a desiccator only	In glass for one month if kept at 2 °C to 6 °C
Chloride	NaCl	1,648 4 ± 0,017	Dry at 105 °C	In polyethylene for three months if kept at 2 °C to 6 °C
Chlorite[a]	$NaClO_2$	≈1,7	Dry in a desiccator only	In glass for one week if kept at 2 °C to 6 °C in the dark

[a] The concentration of the chlorite stock solution shall be determined iodometrically before use (see ISO 10530).

6.10 Standard solutions.

Depending upon the concentrations expected, prepare standard solutions of different anion composition and concentration from the stock solutions (6.9). The risk of changes in concentration caused by interaction with the flask material increases with decreasing anion concentration. Store the standard solutions in polyethylene (PE) flasks. Take into account that sodium chlorite salt can contain up to 20 % sodium chloride. Prepare chlorite standard solutions as described in 6.10.2 to avoid chloride contamination, for example, of the mixed standard solution (6.10.1).

6.10.1 Mixed standard solution of chlorate and chloride.

The mass concentrations of this solution are as follows:

$$\rho_{ClO_3^-,Cl^-} = 10\,mg/l$$

Pipette 1 ml of each of the chlorate and chloride stock solutions (6.9) into a volumetric flask of nominal capacity 100 ml, add 0,1 ml of sodium hydroxide solution (6.4) and fill up to volume with water (6.1).

Prepare the solution on the day of use.

Other mixed standard solutions can be made by respective dilutions of the mixed standard solution.

6.10.2 Chlorite standard solution.

The mass concentration of this solution is as follows:

$$\rho_{ClO_2^-} = 10\,mg/l$$

Pipette 1 ml of chlorite stock solution (6.9) into a volumetric flask of nominal capacity 100 ml, add 0,1 ml of sodium hydroxide solution (6.4) and make up to volume with water (6.1).

Prepare the solution on the day of use.

Other standard solutions can be made by respective dilutions of the chlorite standard solution.

6.11 Anion calibration solutions.

6.11.1 Chlorate, chloride calibration solutions.

Depending on the anion concentration expected, use the stock solutions (6.9) or the mixed standard solution (6.10.1) to prepare 5 to 10 calibration solutions distributed over the expected working range as evenly as possible.

For example, proceed as follow for the range 0,1 mg/l to 1,0 mg/l of ClO_3^-, Cl^-.

Into a series of volumetric flasks of nominal capacity 100 ml, pipette a volume of 1 ml, 2 ml, 3 ml, 4 ml, 5 ml, 6 ml, 7 ml, 8 ml, 9 ml, and 10 ml of the mixed standard solution (6.10.1), add 0,1 ml of sodium hydroxide solution (6.4) and dilute to volume with water (6.1). The concentrations of ClO_3^- and Cl^- in these calibration solutions are 0,1 mg/l, 0,2 mg/l, 0,3 mg/l, 0,4 mg/l, 0,5 mg/l, 0,6 mg/l, 0,7 mg/l, 0,8 mg/l, 0,9 mg/l and 1,0 mg/l, respectively.

Prepare the calibration solutions on the day of use.

6.11.2 Chlorite calibration solutions.

Depending on the anion concentration expected, use the stock solution (6.9) or the chlorite standard solution (6.10.2) to prepare 5 to 10 calibration solutions distributed over the expected working range as evenly as possible.

For example, proceed as follows for the range 0,1 mg/l to 1,0 mg/l ClO_2^-.

Into a series of volumetric flasks of nominal capacity 100 ml, pipette a volume of 1 ml, 2 ml, 3 ml, 4 ml, 5 ml, 6 ml, 7 ml, 8 ml, 9 ml, and 10 ml of the chlorite standard solution (6.10.2), add 0,1 ml of sodium hydroxide solution (6.4) and dilute to volume with water (6.1). The concentrations of ClO_2^- in these calibration solutions are 0,1 mg/l, 0,2 mg/l, 0,3 mg/l, 0,4 mg/l, 0,5 mg/l, 0,6 mg/l, 0,7 mg/l, 0,8 mg/l, 0,9 mg/l and 1,0 mg/l respectively.

Prepare the calibration solutions on the day of use.

6.12 Blank solution.

Fill a volumetric flask of nominal capacity 100 ml up to volume with water (6.1) and add 0,1 ml of sodium hydroxide solution (6.4).

7 Apparatus

Usual laboratory apparatus, and, the following in particular an ion chromatographic system, complying with the quality requirements of Clause 8. In general, it shall consist of the following components (see Figure 1):

a) eluent reservoir;

b) pump, suitable for HPLC;

c) sample injection system incorporating a sample loop (e.g. sample loop of volume 50 µl);

d) precolumn (see 10.3), for example, containing the same resin material as the analytical separator column or those being packed with a macroporous polymer;

e) separator column with the specified separating performance (Clause 8);

f) conductivity detector (with or without a suppressor device assembly) or UV detector (e.g. spectral photometer; 190 nm to 400 nm) or amperometric detector;

g) recording device (e.g. recorder, integrator with printer);

h) cartridges or columns with non-polar phases to be used for sample preparation (e.g. polyvinylpyrrolidone or RP C18[1)] cartridges; see 9.1); the use of RP C18 material is restricted by the pH of the eluent, thus, only RP C18 cartridges should be used, and not columns;

i) cation exchanger in the Ag form (cartridge, 9.2);

j) cation exchanger in the H form (cartridge, 9.2).

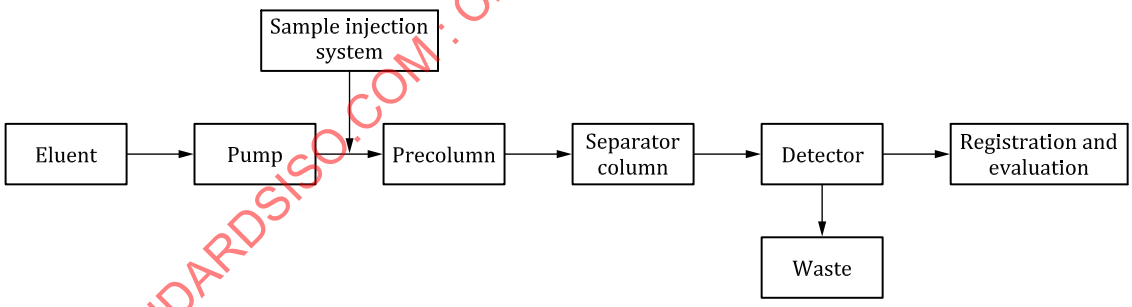


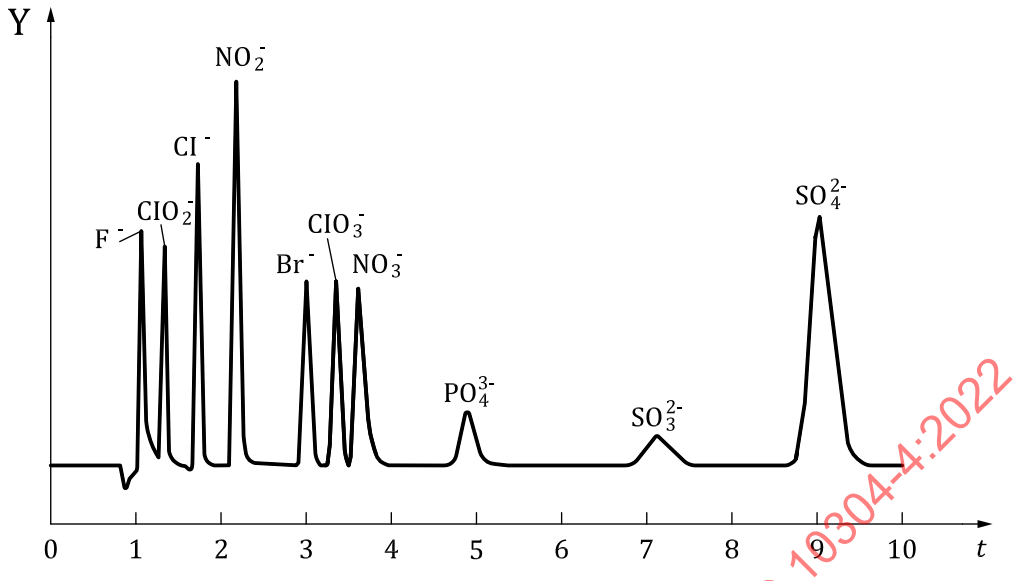
Figure 1 — Schematic representation of an ion chromatography system

8 Quality requirements for the separator column

Separation conditions shall be such that possible interfering anions (fluoride, chlorite, chloride, nitrite, bromide, chlorate and nitrate) at a concentration level of 1 mg/l each (see Figure 2) do not interfere with the anions of interest at a concentration of 1 mg/l.

Regarding chromatograms of samples and standard solutions with higher concentrations, peak resolution R shall not fall below $R = 1{,}3$ [see Formula (1) and (2) and Figure 3].

1) RP C18 is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.



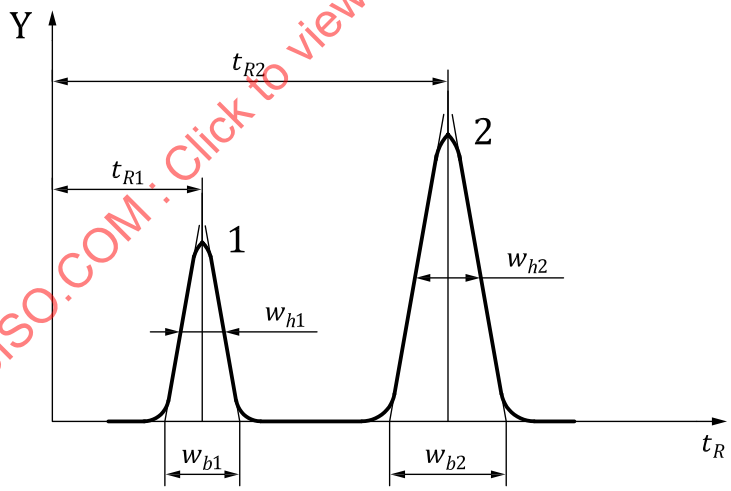
Key

t time, min

Y signal

NOTE Elution sequences and retention times (t_R) can vary, depending on type of column, eluent composition and eluent flow.

Figure 2 — Example of chromatogram from a column conforming to this document



Key

Y signal

1 peak 1

2 peak 2

Figure 3 — Graphical representation of parameters used to calculate peak resolution, R

Calculate the peak resolution, $R_{2,1}$, using Formula (1) or (2).

$$R_{2,1} = 2\frac{(t_{R2} - t_{R1})}{w_{b2} + w_{b1}} \quad (1)$$

$$R_{2,1} = 1{,}18\frac{(t_{R2} - t_{R1})}{w_{h2} + w_{h1}} \quad (2)$$

where

$R_{2,1}$ is the resolution for the peak pair 2,1;

t_{R1} is the retention time, in seconds (s), of the first peak;

t_{R2} is the retention time, in seconds (s), of the second peak;

w_{h1} is the peak width, in seconds (s), on the time axis of the first peak at half the peak height;

w_{h2} is the peak width, in seconds (s), on the time axis of the second peak at half the peak height;

w_{b1} is the peak width, in seconds (s), on the time axis of the first peak;

w_{b2} is the peak width, in seconds (s), on the time axis of the second peak.

NOTE w_{b1} and w_{b2} are the widths of the base of the isosceles triangle constructed representing four times the standard deviation of the Gaussian peak.

9 Sampling and sample pre-treatment

9.1 General requirements

It is important to ensure that the laboratory receives a sample which is truly representative and has not been damaged or changed during transport or storage. Sampling is not part of the method specified in this document.

Drinking water disinfection treatment using chlorine dioxide can cause rapid formation of chlorite and chlorate by excess chlorine dioxide.

If necessary, reasonable precautions should be taken to prevent such risks, for example, stripping of ClO_2 from the sample with an inert gas at a sampling site or before sample injection into the ion chromatograph to minimize possible formation of chlorite and chlorate.

If chlorite or chlorate concentrations at the waterworks site need to be determined, stripping of ClO_2 from the sample, for example with an inert gas, at sampling site should be considered. In most cases, the chlorite and chlorate concentration at consumer's tap is the aim of water analysis. In this case, no stripping of ClO_2 during sampling is necessary.

Use clean polyethylene or glass flasks for sampling.

If necessary, filter the sample after the arrival in the laboratory through a membrane filter (of pore size 0,45 µm) to prevent adsorption of the anions onto particulate matter or conversion of anions by bacterial growth.

NOTE 1 It is not necessary to filter drinking water samples.

If an immediate analysis is not feasible within 48 h, adjust the pH of the samples to a value of 10 ± 0,5 with sodium hydroxide solution (6.4) and stabilize the membrane-filtered sample by cooling it (2 °C to 6 °C) or deep-freezing (–16 °C to –20 °C), provided this procedure does not impair the results. The method of sample pre-treatment shall be noted in the test report.

NOTE 2 Untreated samples can show up to 10 % lower chlorite concentrations.

NOTE 3 The use of pH-electrodes can cause Cl^- contamination.

Prior to injection into the analyser, filter the sample again through a membrane filter (of pore size 0,45 µm) to remove any particulate matter if present.

Avoid contamination of the sample from the membrane (e.g. rinse the membrane with a small amount of the sample itself and discard the first portion of the filtrate).

Waters strongly contaminated with organics can damage the separator column. In this case, it is advisable to dilute the sample and to filter it via a nonpolar phase [e.g. polyvinylpyrrolidone, Clause 7, d), h)] prior to injection (10.3).

Treat blank solutions (6.12) and calibration solutions (6.11) in the same manner as the sample solutions.

Continue with 9.2 if elevated levels of chloride or bromide interfere with the determination of chlorite or chlorate.

9.2 Sample pre-treatment in the case of elevated levels of chloride and bromide

If levels of chloride or bromide are such that peak resolution is no longer acceptable (see Clause 8), reduce their levels by the use of a cation exchanger as follows:

a) dilute the sample if necessary, and run it through a strongly acidic cation exchanger in the Ag form [cartridge, Clause 7, i)] to remove dissolved halides from the sample; before use rinse with water (6.1);

b) run the filtrate through a cation exchanger in the H form [cartridge, Clause 7, j)] to remove dissolved silver ions from the eluate; before use, rinse the cartridge with water (6.1);

c) chromatograph the treated sample as described in Clause 10;

d) treat blank solutions (6.12) and calibration solutions (6.11) in the same manner.

10 Procedure

10.1 General

Set up the ion chromatograph (see Clause 7) according to the instrument manufacturer's instructions (e.g. the instrument is ready for operation as soon as the baseline is stable). Perform the calibration described in 10.2. Measure samples and blank solutions (6.12) as described in 10.3.

10.2 Calibration

Inject the calibration solutions. Identify the peaks for particular anions by comparing the retention times with those of the standard solutions (see 6.11). Take into account the fact that the retention times can be dependent on concentration and matrix. In calculating concentrations, use the characteristic that the area (or height) of the peak (signal) is proportional to the concentration of the anion.

When the analytical system is first evaluated, and at intervals afterwards, establish a calibration function according to ISO 8466-1 for the measurement as follows.

— Prepare calibration solutions as described in 6.11.

— Analyse the calibration solutions chromatographically.

— Use the data obtained to calculate the calibration function. Reject if it is not linear (for linearity criteria, refer to ISO 8466-1).

— Subsequently, check the continuing validity of the established calibration function (see 10.4).

10.3 Measurement of samples using the standard calibration procedure

Filter the sample through a membrane filter of 0,45 µm pore size prior to analyses to remove any particulate matter, if necessary.

Remove remaining chlorine dioxide from the sample, if necessary (9.1). After establishing the calibration function, inject the pre-treated sample (see Clause 9) into the chromatograph and measure the peaks as above (see Clause 10).

In general, the use of a precolumn is strongly recommended, especially for the injection of waters strongly contaminated with organics (9.1), in order to protect the analytical separator column. Two different types of precolumns can be used: those containing the same resin material as the analytical separator column and those packed with a macroporous polymer [see Clause 7, d)].

If the ion concentration of the sample to be analysed exceeds the calibration range, dilute the sample and analyse it. Sometimes, it is necessary to establish a separate calibration function for the lower concentration range.

If matrix interferences are expected, use the method of standard addition to safeguard the results (verify the peaks by comparing the retention times of the spiked sample with those of the original sample). Measure the blank solution (6.12) in the same manner.

10.4 Validity check of the calibration function

In order to verify the continuing validity of the calibration function, measure a minimum of two calibration solutions of different concentrations in the lower and upper parts of the working range. This should take place after the setup procedure of Clause 10 and after each sample series (10.3) at least, but in any case, after 20 measurements or 5 in case of amperometric detection.

Calculate the mass concentrations of the analysed calibration solutions using the inverse calibration function (see Clause 11). The concentrations shall be in the range of the confidence band. If the calibration function is not valid, carry out a new calibration (10.2).

11 Calculation

Calculate the mass concentration in micrograms per litre or milligrams per litre, of the anion in the solution using the peak areas or peak heights according to ISO 8466-1.

Take into account all of the dilution steps.

12 Expression of results

Report the results to a maximum of two significant figures.

EXAMPLE

Chlorate (ClO_3^-) 50 µg/l

Chloride (Cl^-) 35 mg/l

Chlorite (ClO_2^-) 0,15 mg/l

13 Test report

The test report shall contain at least the following information:

a) the test method used, together with a reference to this document, i.e. ISO 10304-4:2022;

b) all information necessary for the complete identification of the sample;

c) expression of the results in accordance with Clause 12;

d) description of sample pre-treatment, if relevant;

e) description of the chromatographic conditions: type of instrument and column, column dimensions, eluent flowrate, type of detector and detector parameters;

f) description of the method used for the evaluation (peak height or peak area);

g) calculation of the results (linear calibration function, method of standard addition);

h) any deviation from this method and information on all circumstances which can have influenced the results;

i) the date of the test.

Annex A
(informative)

Performance data

An interlaboratory trial was organized in Germany in 1996 with laboratories from France and Germany participating. A variety of instruments and other analytical conditions were used which conformed with the quality parameters specified in the method given in this document.

For the description of the sample matrix, see Table A.1.

The statistical data of results are presented in Tables A.2 to A.4.

The coefficients of variation of the procedure V_{X0} (obtained from determined calibration functions analogous to those described in 10.2) are listed in Table A.5. The data came from laboratories participating in the interlaboratory trial in Germany in 1996.

Table A.1 — Description of the sample matrix

Parameter	Sample no.			
	1 and 2	3 and 4	5 and 6	7 and 8
	Sample matrix			
	Synthetic water	Drinking water	River water	Swimming pool water
Base-capacity (mmol/)	0,09	0,39	1,24	0,12
Acid-capacity (mmol/l)	0,22	2,75	3,79	0,29
$\Sigma(Ca^{2+} + Mg^{2+})$ (mmol/l)	0,61	1,3	1,9	1,2
Hydrogencarbonate (mg/l)	13,3	167,3	231,2	17,8
Fluoride (mg/l)	0,12	0,25	0,13	0,05
Chloride (mg/l)	16,1	59,5	68,4	80,2
Nitrate (mg/l)	18,5	4,8	21,8	11,6
Phosphate (mg/l)	0,27	0,05	0,48	0,12
Sulfate (mg/l)	35,1	81,7	58,9	15,7
Bromide (mg/l)	0,21	0,06	0,08	0,005
Chlorate (mg/l)	—	—	—	0,08
Chlorite (mg/l)	—	—	—	—
Sodium (mg/l)	21,5	82,5	82,1	15,1
Potassium (mg/l)	31,7	3,6	4,5	2,5
Magnesium (mg/l)	—	23,0	14,8	12,1
Calcium (mg/l)	—	14,1	52,1	26,3
DOC (mg/l)	0,27	0,52	0,95	0,7